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Term: ptfe membrane and fluorinated solvents

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L1	membrane and "atomic force electronic microscope"	0	L1

END OF SEARCH HISTORY

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Search update 6/25/03

L8: Entry 1 of 2 File: USPT Jun 17, 2003

DOCUMENT-IDENTIFIER: US 6579341 B2
TITLE: Nitrogen gas separation using organic-vapor-resistant membranes

Brief Summary Text (13):
Films or membranes made from fluorinated polymers having a ring structure in the repeat unit are known. For example: 1. U.S. Pat. Nos. 4,897,457 and 4,910,276, both to Asahi Glass, disclose various perfluorinated polymers having repeating units of perfluorinated cyclic ethers, and cite the gas-permeation properties of certain of these, as in column 8, lines 48-60 of U.S. Pat. No. 4,910,276. 2. A paper entitled "A study on perfluoropolymer purification and its application to membrane formation" (V. Arcella et al., Journal of Membrane Science, Vol. 163, pages 203-209 (1999)) discusses the properties of membranes made from a copolymer of tetrafluoroethylene and a dioxole. Gas permeation data for various gases are cited. 3. European Patent Application 0 649 676 A1, to L'Air Liquide, discloses post-treatment of gas separation membranes by applying a layer of fluoropolymer, such as a perfluorinated dioxole, to seal holes or other defects in the membrane surface. 4. U.S. Pat. No. 5,051,114, to Du Pont, discloses gas separation methods using perfluoro-2,2-dimethyl-1,3-dioxole polymer membranes. This patent also discloses comparative data for membranes made from perfluoro(2-methylene-4-methyl-1,3-dioxolane) polymer (Example XI). 5. A paper entitled "Gas and vapor transport properties of amorphous perfluorinated copolymer membranes based on 2,2-bistrifluoromethyl-4,5-difluoro-1,3dioxole/tetrafluoroethylene" (I. Pinnau et al., Journal of Membrane Science, Vol. 109, pages 125-133 (1996)) discusses the free volume and gas permeation properties of fluorinated dioxole/tetrafluoroethylene copolymers compared with substituted acetylene polymers. This reference also shows the susceptibility of this dioxole polymer to plasticization by organic vapors and the loss of selectivity as vapor partial pressure in a gas mixture increases (FIGS. 3 and 4).

Brief Summary Text (26):
Particularly preferred materials for the selective layer of the membrane used to carry out the process of the invention are amorphous homopolymers of perfluorinated dioxole, dioxolanes or cyclic alkyl ethers, or copolymers of these with tetrafluoroethylene. Specific most preferred materials are copolymers having the structure: ##STR1##

Brief Summary Text (27):
where x and y represent the relative proportions of the dioxole and the tetrafluoroethylene blocks, such that x+y=1.

Brief Summary Text (32):
Membranes made from fluorinated dioxoles have been believed previously to behave like conventional membrane materials in suffering from debilitating plasticization in a hydrocarbon-containing environment, to the point that they may even become selective for hydrocarbons over permanent gases even at moderate C.sub.3+ hydrocarbon partial pressures. We have discovered that this is not the case for the membranes taught herein. This unexpected result is achieved because the membranes used in the invention are unusually resistant to plasticization by hydrocarbon vapors.

Brief Summary Text (34):

Besides withstanding exposure during use, their resistance to hydrocarbons enables the membranes and modules to be cleaned with hydrocarbon solvents to remove oils or other organic materials that may have been deposited during operation. This is an additional and beneficial improvement over processes previously available in the art.

Brief Summary Text (42):
The scope of the invention in this aspect is not intended to be limited to any particular gas streams, but to encompass any situation where a gas stream containing nitrogen and a gaseous hydrocarbon is to be separated. The composition of treatable gas streams varies widely, and the individual components may be present in any quantities. Thus, feed gas streams may contain just a few percent nitrogen, or 90% nitrogen or more. The gas may contain a single hydrocarbon component, such as methane, ethylene or a chlorinated solvent, or a mix of numerous hydrocarbons, such as C.sub.1 -C.sub.8 hydrocarbons or heavier. The third component of the gas stream may be a second hydrocarbon, an inert gas, carbon dioxide, water vapor, hydrogen, oxygen or any other component.

Detailed Description Text (17):
As a third non-limiting example, the feed stream may be a gas stream from a petrochemical manufacturing process, comprising a petrochemical feedstock, such as ethylene or propylene, as well as nitrogen and light paraffins or other inert gases. Such streams arise, for example, from manufacture of primary petrochemicals, chemical intermediates, fuels, polymers, agricultural chemicals and the like. A particular example is the manufacture of polyolefins. In a typical polymerization process, the raw polymer product contains significant amounts of unreacted monomer, and may contain small amounts of solvents, catalysts, stabilizers, other hydrocarbons or any other materials, depending on the manufacturing process used.

Detailed Description Text (19):
As a fourth non-limiting example, the feed stream may be an off-gas stream from one of the numerous industrial processes that produce waste streams containing organic vapors in air or nitrogen. Sources of such streams include use of organic solvents in coating, spraying, cleaning, painting, or printing applications of all kinds; organic liquid storage tank vents; chemical manufacturing; and foundry cold boxes using organic catalysts for metal casting. Diverse organic vapors may be present in streams of these types, including, for example, halogenated solvents, alkyl amines, ketones or alcohols.

Detailed Description Text (20):
The process of the invention may be used to permeate the oxygen and nitrogen, leaving an organic-enriched residue stream from which the organic solvent can be recovered for reuse. As it relates to separation of nitrogen from larger and more complex organic molecules, such as halogenated solvents or amines, the process of the invention can often provide very high mixed-gas selectivities, such as 20, 40, 50, 100 or higher, depending on the nature of the organic compound and the process conditions.

Detailed Description Text (34):
The ring structure within the repeat units may be aromatic or non-aromatic, and may contain other atoms than carbon, such as oxygen atoms. Preferred polymers for the selective layer of the membrane are formed from fluorinated monomers of (i) dioxoles, which are five-member rings of the form ##STR3##

Detailed Description Text (36):
Not all polymers within the above structural definitions and preferences are suitable for use as membrane selective layers in the invention. For example, certain of the polymers and copolymers of perfluoro-2,2-dimethyl-1,3-dioxole reported in U.S. Pat. No. 5,051,114 have been shown to be susceptible to plasticization to the point of switching from being selective for nitrogen over hydrocarbons to being selective for hydrocarbons over nitrogen as the hydrocarbon partial pressure increases.

Detailed Description Text (37):
These polymers are, however, characterized by very high fractional free volume

within the polymer, typically above 0.3. For example, a paper by A. Yu. Alentiev et al, "High transport parameters and free volume of perfluorodioxole copolymers", Journal of Membrane Science, Vol. 126, pages 123-132 (1997) reports fractional free volumes of 0.32 and 0.37 for two grades of perfluoro-2,2-dimethyl-1,3-dioxole copolymers (Table 1, page 125).

Detailed Description Text (47):
The polymers may be homopolymers of the repeating units of fluorinated cyclic structures defined above. Optionally, they may be copolymers of such repeat units with other polymerizable repeat units. For preference, these other repeat units should be at least partially fluorinated, and most preferably heavily fluorinated or perfluorinated. A number of suitable materials are known, for example, fluorinated ethers, ethylene and propylene. Particularly when perfluorinated, homopolymers made from these materials, such as polytetrafluoroethylene (PTFE) and the like, are very resistant to plasticization. However, they tend to be crystalline or semi-crystalline and to have gas permeabilities too low for any useful separation application. As constituents of copolymers with the fluorinated ring structures defined above, however, they can produce materials that combine amorphous structure, good permeability and good resistance to plasticization. Copolymers that include tetrafluoroethylene units are particularly preferred. Other specific examples of copolymers that are suitable are polyhexafluoropropylene and chlorofluoro ethylenes and propylene.

Detailed Description Text (48):
Specific most preferred materials are copolymers of tetrafluoroethylene with 2,2,4-trifluoro-5-trifluoromethoxy-1,3-dioxole having the structure: ##STR4##

Detailed Description Text (49):
where x and y represent the relative proportions of the dioxole and the tetrafluoroethylene blocks, such that x+y=1.

Detailed Description Text (50):
Such materials are available commercially from Ausimont S.p.A., of Milan, Italy under the trade name Hyflon.RTM. AD. Different grades are available varying in proportions of the dioxole and tetrafluoroethylene units, with fluorine:carbon ratios of between 1.5 and 2, depending on the mix of repeat units. For example, grade Hyflon AD60 contains a 60:40 ratio of dioxole to tetrafluoroethylene units, has a fractional free volume of 0.23, a density of 1.93 g/cm.sup.3 and a glass transition temperature of 121.degree. C., and grade Hyflon AD80 contains an 80:20 ratio of dioxole to tetrafluoroethylene units, has a fractional free volume of 0.23, a density of 1.92 g/cm.sup.3 and a glass transition temperature of 134.degree. C.

Detailed Description Text (60):
The polymer chosen for the selective layer can be used to form films or membranes by any convenient technique known in the art, and may take diverse forms. Because the polymers are glassy and rigid, an unsupported film, tube or fiber of the polymer may be usable in principle as a single-layer membrane. In this case, our preferred method of manufacture is to prepare a solution of the polymer in a perfluorinated solvent and to cast the solution onto a glass plate or a removable or non-removable backing web, according to general casting procedures that are well known in the art. The resulting flat-sheet membrane films may be dried under ambient conditions, at elevated temperature, or under vacuum as desired to produce thin film membranes.

Detailed Description Text (67):
Preferred materials for the gutter layer are fluorinated or perfluorinated, to maintain high chemical resistance through the membrane structure, and of very high permeability. Particularly preferred for the gutter layer, although they are unsuitable for the selective layer, are the perfluorinated dioxole polymers and copolymers of U.S. Pat. No. 5,051,114 referred to above, having fractional free volume greater than 0.3 and extraordinarily high permeability, such as copolymers of perfluoro-2,2-dimethyl-1,3dioxole and tetrafluoroethylene, available commercially as Teflon.RTM. AF from DuPont Fluoroproducts of Wilmington, Del. Such materials, or any others of good chemical resistance that provide protection for the selective layer without contributing significant resistance to gas transport, are also suitable as sealing layers.

Detailed Description Text (68):
Multiple selective layers may also be used. In particular, depending on the properties of the material used for the asymmetric support membrane and the porosity of the skin, the skin layer of the support may also act as a selective layer to provide a contribution to the overall selectivity of the composite.

Detailed Description Text (70):
Once formed, the membranes exhibit a combination of good mechanical properties, thermal stability, and high chemical resistance. The fluorocarbon polymers that form the selective layer are typically insoluble except in perfluorinated solvents and are resistant to acids, alkalis, oils, low-molecular-weight esters, ethers and ketones, aliphatic and aromatic hydrocarbons, halogenated compounds and oxidizing agents, making them suitable for use not only in the presence of C.sub.3+ hydrocarbons, but in many other hostile environments.

Detailed Description Text (74):
An important consideration is the effect of hydrocarbons, particularly C.sub.3+ hydrocarbons, in the feed stream. Unlike prior art membranes, the membranes of the invention can maintain useful gas/hydrocarbon separation performance, in terms of transmembrane gas flux and selectivity, when exposed to high concentrations of such organics, even when the gas mixture is close to saturation with these compounds. This is true with respect to a broad range of hydrocarbons, including paraffins, olefins, aromatics, such as benzene, toluene and xylenes (BTEX), alcohols and chlorinated compounds. These properties are different from those reported in the literature for dioxole membranes, as well as obtained with prior art conventional membrane materials, such as cellulose acetate, polysulfone, or polyimides that are not perfluorinated.

Detailed Description Text (77):
The ability of the membranes to withstand exposure to organic compounds means that the membrane modules may be cleaned with organic solvents. This is a very useful property. Industrial gas streams often contain small amounts of entrained oils and other relatively heavy organic components, which may enter the separation system and become trapped in the modules, fouling the membrane surface. Even if the membranes themselves are not damaged, the performance may be adversely affected by the build-up of such materials. One attractive feature of the present invention is that the modules can be cleaned periodically or as required by flushing with organic solvents to remove such contaminants.

Detailed Description Text (90):
Asymmetric, microporous poly(vinylidene fluoride) [PVDF] support membranes were prepared. Composite membranes were prepared using the following coating solutions: 1 wt % copolymer solution of 40% tetrafluoroethylene/60% 2,2,4-trifluoro-5-trifluoromethoxy-1,3-dioxole (Hyflon.RTM. AD60), (Ausimont, Italy), in a perfluorinated solvent (Fluorinert FC-84), (3M, St. Paul, Minn.). 1 wt % copolymer solution of 20% tetrafluoroethylene/80% 2,2,4-trifluoro-5-trifluoromethoxy-1,3-dioxole (Hyflon.RTM. AD80), (Ausimont, Italy), in FC-84 solvent. 1 wt % polyperfluoro (alkenyl vinyl ether) (Cytop.RTM.), (Asahi Glass, Japan), in FC-84 solvent.

Detailed Description Text (98):
Solvent Resistance of Hyflon.RTM. AD60 compared to Polysulfone

Detailed Description Text (99):
Experiments were carried out to determine the stability of a Hyflon.RTM. AD60 membrane in the presence of hydrocarbon solvents. Samples of a Hyflon.RTM. AD60 membrane were tested in a permeation test-cell as in Example 1. The fluxes were measured and the selectivities calculated. The membrane stamps were then immersed in liquid toluene or hexane. After one week, the membranes were removed from the hydrocarbon liquid, dried at ambient temperature, and retested in the gas permeation test-cell. A polysulfone (PSF) asymmetric membrane, typically used in hydrogen separation processes, was also tested for comparison. The permeation properties of the Hyflon.RTM. AD60 and polysulfone membranes before and after exposure to the hydrocarbon solvent are summarized in Table 4.

Detailed Description Text (100):
As can be seen, the polysulfone membranes could not withstand exposure to toluene, and their hydrogen/methane selectivity declined by half after exposure to hexane. With respect to nitrogen/methane, the selectivity remained marginal, but tipped in favor of methane. In contrast, the dioxole copolymer Hyflon.RTM. membranes, although they exhibited higher fluxes for all gases for which they were tested after soaking in liquid hydrocarbons, retained their hydrogen/methane selectivity.

Detailed Description Text (105):
Asymmetric, microporous poly(vinylidene fluoride) [PVDF] support membranes were prepared. Composite membranes were prepared by dip-coating the support membranes three times in a solution of 1 wt % 2,2-bistrifluoromethyl-4,5-difluoro-1,3-dioxole/tetrafluoroethylene copolymer [Teflon.RTM. AF2400] solution in FC-84 solvent at 1 ft/min coating speed, then dried in an oven at 60.degree. C. for 10 minutes. The resulting membranes had a selective layer thickness of 4 .mu.m.

Detailed Description Text (142):
Asymmetric, microporous poly(vinylidene fluoride) [PVDF] support membranes were prepared. Composite membranes were prepared using a coating solution of 1 wt % of the polyimide poly(3,4,3',4'-biphenyltetracarboxylic dianhydride-2,4,6-m-phenylenediamine) [BPDA-TMPD] in a hexafluoropropanol/chloroform solvent.

CLAIMS:

9. The process of claim 1, wherein the polymer is formed from a monomer selected from the group consisting of fluorinated dioxoles, fluorinated dioxolanes and fluorinated cyclically polymerizable alkyl ethers.

11. The process of claim 1, wherein the repeat unit is 2,2,4-trifluoro-5-trifluoromethoxy-1,3-dioxole.

14. The process of claim 1, wherein the polymer has the formula: ##STR9##

where x and y represent the relative proportions of the dioxole and the tetrafluoroethylene blocks, such that x+y=1.

28. The process of claim 1, further comprising removing a contaminant material that has been brought into a module housing the separation membrane during operation of steps (a) through (d) by: (e) discontinuing steps (a) through (d); and (f) flushing the module with an organic solvent.

37. The process of claim 29, wherein the polymer is formed from a monomer selected from the group consisting of fluorinated dioxoles, fluorinated dioxolanes and fluorinated cyclically polymerizable alkyl ethers.

39. The process of claim 29, wherein the repeat unit is 2,2,4-trifluoro-5-trifluoromethoxy-1,3-dioxole.

42. The process of claim 29, wherein the polymer has the formula: ##STR11##

where x and y represent the relative proportions of the dioxole and the tetrafluoroethylene blocks, such that x+y=1.

56. The process of claim 29, further comprising removing a contaminant material that has been brought into a module housing the separation membrane during operation of steps (a) through (d) by: (e) discontinuing steps (a) through (d); and (f) flushing the module with an organic solvent.

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☐ 1. Document ID: US 6072020 A

L11: Entry 1 of 1 File: USPT Jun 6, 2000

US-PAT-NO: 6072020
DOCUMENT-IDENTIFIER: US 6072020 A

TITLE: Fluorinated polymer purification

DATE-ISSUED: June 6, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Arcella; Vincenzo	Novara			IT
Brinati; Giulio	Milan			IT
Maccone; Patrizia	Milan			IT

US-CL-CURRENT: 528/176

Full Title CIT.1 REV.1 CLS.1 REF.1 SEQ.1 ATT.1 RAW.1

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L12: Entry 1 of 6 File: USPT Jun 25, 2003

DOCUMENT-IDENTIFIER: US 6582496 B1
TITLE: Hollow fiber membrane contactor

Brief Summary Text (9):
Manufacturers produce membranes from a variety of materials, the most general class being synthetic polymers. An important class of synthetic polymers are thermoplastic polymers, which can be flowed and molded when heated and recover their original solid properties when cooled. As the conditions of the application to which the membrane is being used become more severe, the materials that can be used become limited. For example, the organic solvent-based solutions used for wafer coating in the microelectronics industry will dissolve or swell and weaken most common polymeric membranes. The high temperature stripping baths in the same industry consist of highly acid and oxidative compounds, which will destroy membranes made of common polymers. Perfluorinated thermoplastic polymers such as poly(tetrafluoroethylene-co-perfluoro(alkylvinylether)) (poly(PTFE-CO-PFVAE)) or poly(tetrafluoroethylene-co-hexafluoropropylene) (FEP) are not adversely affected by severe conditions of use, so that membranes made from these polymers would have a decided advantage over ultrafiltration membranes made from less chemically and thermally stable polymers. These thermoplastic polymers have advantages over (poly(tetrafluoroethylene) (PTFE), which is not a thermoplastic, in that they can be molded or shaped in standard type processes, such as extrusion. Perfluorinated thermoplastic hollow fiber membranes can be produced at smaller diameters than possible with PTFE. Fibers with smaller diameters, for example, in the range of about 350 micron outer diameter to about 1450 micron outer diameter, can be fabricated into contactors having high membrane surface area to contactor volume ratios. This attribute is useful for producing compact equipment, which are useful in applications where space is at a premium, such as in semiconductor manufacturing plants.

Brief Summary Text (10):
Being chemically inert, the Poly (PTFE-CO-PFVAE) and FEP polymers are difficult to form into membranes using typical solution casting methods as they are difficult to dissolve in the normal solvents. They can be made into membranes using the Thermally Induced Phase Separation (TIPS) process. In one example of the TIPS process a polymer and organic liquid are mixed and heated in an extruder to a temperature at which the polymer dissolves. A membrane is shaped by extrusion through an extrusion die, and the extruded membrane is cooled to form a gel. During cooling the polymer solution temperature is reduced to below the upper critical solution temperature. This is the temperature at or below which two phases form from the homogeneous heated solution, one phase primarily polymer, the other primarily solvent. If done properly, the solvent rich phase forms a continuous interconnecting porosity. The solvent rich phase is then extracted and the membrane dried.

Brief Summary Text (20):
In U.S. Pat. No. 5,464,480, preferably, the bubbles that are provided by the diffuser are initially about 25 to about 40 microns in diameter. The gas diffuser preferably is made from a mixture of polytetrafluoroethylene (PTFE) and perfluoroalkoxylvinylether. By varying the temperature and pressure under which the mixture is prepared by methods known in the art, both porous and nonporous members are formed. The impermeable and permeable members are preferably comprised of about 95% PTFE and about 5% perfluoroalkoxylvinylether. The permeable member and the impermeable member may be joined by any number of methods as long as the result is a

composite member that will not come apart under the stresses in the tank. Preferably, the members are heat sealed together, essentially melting or fusing the members together using carbon-carbon bonds. Once the permeable member is formed, a trench is bored out of the PTFE in the top portion of the member. The resulting diffuser has on the order of about 100,000 pores of a size of about 25 to about 40 microns in diameter through which gas may permeate into the treatment tank. The use of the trench in the diffuser allows the gas to diffuse into the tank as very fine bubbles. In applications for the semiconductor manufacturing industry, a device that supplied homogeneous bubble free ozone dissolved in ultrapure water would provide more efficient oxidation reactions because the reaction would not be localized at the bubbles. The more homogeneous solution would provide for a more uniform cleaning reaction. Furthermore, the high surface area to volume ratio inherent in hollow fiber devices would give a compact system, suitable for semiconductor operations.

Brief Summary Text (24):
An advantage for contacting applications is that the very low surface tension of these perfluorinated polymers allows use with low surface tension liquids. For example, highly corrosive developers used in the semiconductor manufacturing industry may contain surface tension reducing additives, such as surfactants. These developers could not be degassed with typical microporous membranes because the liquid would intrude the pores at the pressures used and permeate, causing solution loss and excess evaporation. In addition, liquid filling the pores would greatly add to the mass transfer resistance of gas transport. U.S. Pat. No. 5,749,941 describes how conventional hollow fiber membranes of polypropylene or polyethylene cannot be used in carbon dioxide or hydrogen sulfide absorption into aqueous solutions containing an organic solvent without the use of a solution additive to prevent leakage. While polytetrafluoroethylene (PTFE) membranes would work in these applications, presumably because of their lower surface tension, they are difficult to process into hollow fibers. The membranes of the present invention are made from polymers having similar surface tension properties to PTFE and are more readily manufactured into small diameter hollow fiber membranes.

Brief Summary Text (25):
WO 9853894 describes a process of forming compact, high flux, fouling resistant gas filters by coating continuous ultra thin layer of non-porous gas permeable polymer over filter surface by contacting one side of microporous substrate with dilute coating solution of polymer, preferably amorphous copolymer of perfluoro-2,2-dimethyl-1,3-dioxole, which is hydrophobic and oleophobic. Substrate pore size filters polymer from solution as solvent flows through, leaving ultra thin layer of polymer. Process is useful to coat sheet and hollow fiber substrates, particularly multiple hollow fibers assembled in modules. These membranes have been described as useful for contactor applications. (S. Nemser, Paper presented at 1998 North American Membrane Society Meeting.) This method requires a separate and complex coating step to produce a non-porous contactor fiber. Furthermore, a fully perfluorinated thermoplastic contactor is not described.

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1. Document ID: US 6582496 B1

L12: Entry 1 of 6 File: USPT Jun 24, 2003

US-PAT-NO: 6582496
DOCUMENT-IDENTIFIER: US 6582496 B1

TITLE: Hollow fiber membrane contactor

DATE-ISSUED: June 24, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cheng; Kwok-Shun	Nashua	NH		
Doh; Cha P.	Sudbury	MA		

US-CL-CURRENT: 95/46; 95/45, 96/14, 96/6, 96/8

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw Desc | Image

2. Document ID: US 6579342 B2

L12: Entry 2 of 6 File: USPT Jun 17, 2003

US-PAT-NO: 6579342
DOCUMENT-IDENTIFIER: US 6579342 B2

TITLE: Oleophobic membrane materials by oligomer polymerization for filter venting applications

DATE-ISSUED: June 17, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wang; I-fan	San Diego	CA		
Palpallatoc; Jeff	San Diego	CA		

US-CL-CURRENT: 95/46; 55/524, 55/DIG.5, 96/13, 96/14, 96/224, 96/225, 96/6

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw Desc | Image

3. Document ID: US 6569341 B2

L12: Entry 3 of 6 File: USPT May 27, 2003

US-PAT-NO: 6569341
DOCUMENT-IDENTIFIER: US 6569341 B2

TITLE: Osmotic distillation process

DATE-ISSUED: May 27, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Bowser; John J.	Newark	DE		

US-CL-CURRENT: 210/640; 210/500.36, 210/634, 210/644, 426/14, 426/239, 426/425, 95/46

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments KWIC
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4. Document ID: US 6521012 B2

L12: Entry 4 of 6 File: USPT Feb 18, 2003

US-PAT-NO: 6521012
DOCUMENT-IDENTIFIER: US 6521012 B2

TITLE: Oleophobic coated membranes

DATE-ISSUED: February 18, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Lamon; Steven	San Diego	CA		
McDonogh; Richard	San Diego	CA		

US-CL-CURRENT: 55/524; 427/255.6, 427/385.5, 428/98, 55/DIG.5, 96/12

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments KWIC
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5. Document ID: US 6299777 B1

L12: Entry 5 of 6 File: USPT Oct 9, 2001

US-PAT-NO: 6299777
DOCUMENT-IDENTIFIER: US 6299777 B1
**** See image for Certificate of Correction ****

TITLE: Osmotic distillation process

DATE-ISSUED: October 9, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Bowser; John J.	Newark	DE		

US-CL-CURRENT: 210/640; 210/634, 210/644, 426/239, 426/425

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments KWIC
Draw. Desc | Image

6. Document ID: US 6072020 A

L12: Entry 6 of 6 File: USPT Jun 6, 2000

US-PAT-NO: 6072020
DOCUMENT-IDENTIFIER: US 6072020 A

TITLE: Fluorinated polymer purification

DATE-ISSUED: June 6, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Arcella; Vincenzo	Novara			IT
Brinati; Giulio	Milan			IT
Maccone; Patrizia	Milan			IT

US-CL-CURRENT: 528/176

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments KWIC
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L12: Entry 2 of 6 File: USPT Jun 17, 2003

DOCUMENT-IDENTIFIER: US 6579342 B2
TITLE: Oleophobic membrane materials by oligomer polymerization for filter venting applications

Brief Summary Text (5):
Polytetrafluoroethylene (PTFE) has been the most commonly used material in filters for gas venting. PTFE is chemically and biologically inert, has high stability, and is hydrophobic. PTFE filters therefore allow gases to be selectively vented while being impervious to liquid water.

Brief Summary Text (9):
The oleophobicity of PTFE can be improved by impregnating or coextruding the PTFE with siloxanes (for example, U.S. Pat. No. 4,764,560), fluorinated urethane (U.S. Pat. No. 5,286,279), or perfluoro-2,2-dimethyl-1,3-dioxole (U.S. Pat. No. 5,116,650). Although the oil resistance of the PTFE is improved, the treated PTFE membranes are expensive, and air permeability remains fairly low.

Brief Summary Text (48):
In a particularly preferred embodiment, the polymer is a sulfone polymer, such as polysulfone, polyethersulfone (PES), or polyarylsulfone. Other suitable polymers include fluorinated polymers such as polyvinylidene fluoride (PVDF), polyolefins including polyethylene and polypropylene, polytetrafluoroethylene (PTFE or Teflon.TM.), poly(tetrafluoroethylene-co-ethylene) (ECTFE or Halar.TM.), acrylic copolymers, polyamides or nylons, polyesters, polyurethanes, polycarbonates, polystyrenes, polyethylene-polyvinyl chloride, polyacrylonitrile, cellulose, and mixtures or combinations thereof.

Brief Summary Text (52):
The membranes of preferred embodiments also have a porous supporting structure between the two sides of the membrane. The nature of the porous supporting structure of a membrane may depend on the composition of the casting dope and the coagulation bath. The supporting structure can include closed cells, open cells of substantially the same pore size from one side of the membrane to the other, open cells with a gradation of pore sizes from one side of the membrane to the other, or finger-type structures, generally referred to as "macrovoids." Macrovoids typically vary substantially in size from the surrounding porosity, and generally do not communicate with surface pores. In a preferred embodiment, the porous supporting structure includes a network of structural surfaces capable of contacting a filter stream, defined herein as including any fluid substance, including liquids and gases, that passes through the membrane via the porous supporting structure.

Brief Summary Text (54):
Membranes that are suitable for grafting in accordance with the present invention include virtually any formed hydrophobic or hydrophilic polymer membranes. Suitable membranes may typically have pore diameters from about 0.001 .mu.m to about 50 .mu.m or greater, preferably from about 0.01 .mu.m to about 50 .mu.m, on the skin side of the membrane. Membranes that are suitable for coating in accordance with the preferred embodiments include, for example, membranes that typically possess porosities characteristic of microfiltration membranes. Microfiltration membranes typically possess pore diameters of from at least about 0.01 or 0.05 .mu.m to about 5, 8, 10 or 20 .mu.m on the skin side of the membrane. Membranes within the ultrafiltration range may also be grafted according to preferred embodiments.

Ultrafiltration membranes typically possess molecular weight cutoffs of from about 10,000 Daltons to about 1,000,000 Daltons and may have pore diameters typically from about 0.001 .mu.m to about 0.050 .mu.m on the skin side of the membrane.

Brief Summary Text (55):
Particularly preferred membranes before post treatment, such as crosslinking or grafting, include the highly asymmetric polyethersulfone membranes disclosed in U.S. Pat. No. 5,886,059 (incorporated herein by reference in its entirety). In typical highly asymmetric PES membranes, one side of the PES membrane is a skin face having relatively small diameter pores while the opposite or dull face of the membrane has relatively large diameter pores. The difference in porosity between the skin face and the opposite face is typically from at least about 1:2, 1:5, or 1:10 to about 1:20, 1:50, 1:100, 1:200 or 1:10,000. Preferably, the difference in porosity is from about 1:2 to about 1:10,000. More preferably, the difference in porosity is from about 1:2 to about 1:200. Most preferably, the difference in porosity is from about 1:5 to about 1:20. In addition, such membranes generally have a gradual slope of pore size from the skin face to the opposite face. Thus, during filtration, larger particles can enter the membrane through the larger pores, but do not exit through the smaller pores. Because the larger particles become lodged just within the outer surface, the membranes made by the methods included herein are not easily clogged with large particles.

Brief Summary Text (59):
Composite membranes may be prepared using lamination techniques. In lamination, sheets are layered together in a stack, optionally with one or more adhesive materials placed between the sheets to facilitate binding and lamination of the sheets to each other, and the stack is laminated into an integral composite membrane under application of heat and/or pressure. A different approach to making composite membranes is to cast or form one membrane layer in situ on top of another layer. The base layer may be a fibrous backing material or it may be a membrane. The composites may include, for example, composites having different layers of any of the foregoing media, composites having multiple layers of the same medium, or composites having layers of the same medium, but of different pore sizes, porosities, geometries, orientations, and the like. The composite may be formed either before or after a membrane component is coated with a fluorosulfone oligomer according to a preferred embodiment.

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L17: Entry 3 of 5 File: USPT Nov 30, 1993

DOCUMENT-IDENTIFIER: US 5266206 A
TITLE: Process for recovering organic components from liquid streams

Brief Summary Text (10):
Contamination of industrial effluent waters with dissolved organic solvents, such as methanol, ethanol, methyl ethyl ketone, phenol, benzene, toluene, and trichloroethane, is an important environmental problem. These solvents make water unfit for reuse or direct discharge to municipal sewers, and are difficult to remove, even at low concentrations. Commonly used methods for removing volatile organic compounds include air stripping, biological treatment, carbon adsorption and incineration. Air stripping, in which water is circulated against a current of air in a contacting tower, is the least expensive process. However, air stripping merely exchanges water pollution for air pollution. Air stripping is therefore seldom used if the solvent concentration exceeds 0.1%, and even then only for small streams where the total organic emission is less than 10-100 lb/day. Carbon adsorption, a principal effluent treatment technology, can only be used efficiently for very dilute streams, typically 1,000 ppm or less, and more usually 100 ppm or less. At these very low concentrations, carbon adsorption is a preferred technique, because the size of the plant scales in proportion to the amount of solvent removed. Thus, when the solvent concentration is very low, the amount of wastewater that can be treated by a small carbon adsorption system is high. However, once the feed solution concentration exceeds 1,000 ppm, carbon adsorption systems become very large per gallon of wastewater treated. Also, carbon adsorption systems cannot handle some chlorinated and fluorinated solvents, and generate secondary waste, in the form of spent contaminated carbon that may be sent to landfills. Biological treatment systems work well only for organics that can be fully metabolized by the biomass, and where the process is not compromised by high or fluctuating solvent concentrations. At the high concentration end of the scale, incineration is reliable and effective for very concentrated streams, where the heat value of the solvent reduces the amount of supplemental fuel required. Typically, incineration is impossibly expensive at concentrations below 5%.

Detailed Description Text (10):
Alternatively, the decantation step can be carried out first, and the waste stream from the decanter can be treated by pervaporation. Pervaporation is a low-pressure membrane process that can be used to separate components of differing volatilities from solutions. A permselective membrane performs the separation. The membranes for use in the process of the invention should be selectively permeable to organics over water, or to the organic to be separated over other organics. Some membrane materials, particularly rubbery polymers, possess an intrinsically high selectivity for organic solvents over water and can therefore be used in the pervaporation step of the invention. Preferred permselective membrane materials include rubbery non-crystalline polymers, with glass transition temperatures below the normal operating temperature of the system. Thermoplastic elastomers are also useful. These polymers combine hard and soft segments or domains in the polymer structure. Provided the soft segments are rubbery at the temperature and operating conditions of the invention, polymers of this type could make suitable membranes for use in the invention. Polymers that may be used include, but are not limited to, nitrile rubber, neoprene, polydimethylsiloxane (silicone rubber), chlorosulfonated polyethylene, polysilicone-carbonate copolymers, fluororelastomers, plasticized polyvinylchloride, polyurethane, cis-polybutadiene, cis-polyisoprene, poly(butene-1), polystyrene-butadiene copolymers, styrene/butadiene/styrene block

copolymers, styrene/ethylene/butylene block copolymers, thermoplastic polyolefin elastomers, polyesteramides, and block copolymers of polyethers and polyesters. Most preferred membrane materials are polystyrene-butadiene block copolymers or silicone rubbers. The membrane may take the form of a homogeneous membrane, an asymmetric membrane, a multilayer composite membrane, a matrix incorporating a gel or liquid layer, or any other form known in the art. A particularly preferred embodiment of the invention employs a multilayer membrane comprising a microporous support onto which is coated the ultrathin permselective layer. The microporous support membrane should have a flow resistance that is very small compared to the permselective layer. Preferably the pores in the skin layer should be less than 1 micron in diameter, to enable it to be coated with a defect-free permselective layer. A preferred support membrane is an asymmetric Loeb-Sourirajan type membrane, which consists of a relatively open, porous substrate with a thin, dense, finely porous skin layer. The support membrane should resist the solvents used in applying the permselective layer. Polymers that may be used to make the microporous support membrane include, for example, polysulfone, polyimide, polyvinylidene fluoride, polyamides, polypropylene or polytetrafluoroethylene. Other polymers suitable for making support membranes are listed in U.S. Pat. No. 4,230,463, column 21, lines 8 through 23, incorporated herein by reference. Commercially available asymmetric ultrafiltration membranes, for example, NTU.RTM. 4220 (crosslinked polyimide), or NTU.RTM. 3050 (polysulfone) from Nitto Electric Industrial Company, Osaka, Japan, could also be used as the support membrane. The thickness is not critical, since the permeability of the support is high compared to that of the permselective layer. A typical support would have a thickness in the range about 100 to 300 microns.

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L20: Entry 1 of 16 File: USPT Jun 5, 2001

DOCUMENT-IDENTIFIER: US 6242135 B1

**** See image for Certificate of Correction ****

TITLE: Solid electrolyte composite for electrochemical reaction apparatus

Detailed Description Text (46):

The ion-exchange resins used in the invention can be prepared by general polymerization techniques developed for homo and copolymerizations of fluorinated ethylenes, particularly those employed for tetrafluoroethylene which are described in the literature. Nonaqueous techniques for preparing the copolymers include that of U.S. Pat. No. 3,041,317, that is, by the polymerization of a mixture of the major monomer therein, such as tetrafluoroethylene, and a fluorinated ethylene containing a sulfonyl fluoride group in the presence of a free radical initiator, preferably a perfluorocarbon peroxide or azo compound, at a temperature in the range 0-200.degree. C. and at pressures in the range of 105 to 2.times.107 Pascals (1-200 Atm.) or higher. The nonaqueous polymerization may, if desired, be carried out in the presence of a fluorinated solvent. Suitable fluorinated solvents are inert, liquid, perfluorinated hydrocarbons, such as perfluoromethylcyclohexane, perfluorodimethylcyclobutane, perfluorooctane, perfluorobenzene and the like, and inert, liquid chloro-fluorocarbons such as 1,1,2-trichloro-1,2-2-trifluoroethane, and the like. Aqueous techniques for preparing such copolymers include contacting the monomers with an aqueous medium containing a free-radical initiator to obtain a slurry of polymer particles in non-water-wet or granular form, as disclosed in U.S. Pat. No. 2,393,967, or contacting the monomers with an aqueous medium containing both a free-radical initiator and a telogenically inactive dispersing agent, to obtain an aqueous colloidal dispersion of polymer particles, and coagulating the dispersion, as disclosed, for example, in U.S. Pat. No. 2,559,752 and U.S. Pat. No. 2,593,583.

Detailed Description Text (59):

Alternatively, the functional material can be introduced into the micropores of the polymeric sheet by imbibing the material into the pores and then heating to remove the imbibing solvent. When the microporous polymeric sheet is polytetrafluoroethylene (PTFE), the functional material can be added by mixing a particulate filler with a PTFE aqueous dispersion; cocoagulating the filler and the PTFE; lubricating the filled PTFE with lubricant; paste extruding and optionally calendering to form a film; and expanding the film by stretching it so as to form a porous PTFE membrane having the filler distributed therein

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L20: Entry 5 of 16 File: USPT May 9, 2000

DOCUMENT-IDENTIFIER: US 6060530 A
TITLE: Process for manufacture of a porous polymer by use of a porogen

Brief Summary Text (5):
Polymers based on perfluoropolyethers, in general, have many unique and desirable properties. These include resistance to fouling by proteinaceous and other materials, outstanding flexibility, transparency, high resistance to temperature extremes, and exceptional chemical and oxidation resistance. These properties would make perfluoropolyether based polymers particularly suitable for a variety of applications and would be particularly suited for use as membranes if methods were available for the economic introduction of porosity. Indeed, there has been a long-felt need for membrane materials with the above attributes. Polytetrafluoroethylene-based (PTFE) membrane materials provide a partial solution to this need. However, unlike perfluoropolyether-based polymers, which can be readily cured and formed into articles by in-situ polymerisation, PTFE-based materials suffer from the disadvantage of being difficult to fabricate and manufacture into articles. In addition, stretching processes such as those described in U.S. Pat. No. 3,953,566 (Gore) give a somewhat limited range of size and shape of the porosities and are difficult to control.

Brief Summary Text (121):
By way of example, the process can be used in the manufacture of articles, such as ophthalmic devices, preferably contact lenses. In such a case the appropriate quantities of polymerizable monomers, solvent (if required) and photoinitiator are mixed together to form a polymerization mixture. The polymerization mixture is then flushed with nitrogen and the required quantity dispensed into the concave half of a polypropylene mould. The mould is closed and clamped and the assembly is placed into a UV irradiation cabinet equipped with UV lamps. The irradiation is performed for the required time and then the halves of the mould are separated. The polymerized lens is extracted in an appropriate solvent (for example, an isopropyl or tert-butylacetate/fluorinated solvent mixture). The solvent is then extensively exchanged with an alcohol (for example, isopropyl alcohol) and subsequently with saline to yield the product lens.

Brief Summary Text (129):
A generally applicable procedure for handling the porous polymers, once polymerized, is e.g. as follows: The polymers are removed from the mold and placed through a general extraction and drying procedure to remove any unpolymerised components. This procedure consists of a 4 h soaking in a fluorinated solvent (PF5060 from 3M Corporation), then 16 hr immersion in isopropyl acetate and subsequent immersion for 4 h in isopropyl alcohol. After drying in vacuo the polymer takes on a white colour. When the white polymer undergoes a graded solvent change from ethanol, 75% ethanol/water, 50% ethanol/water, 25% ethanol/water, then pure water or saline, it becomes transparent. The graded solvent change has the effect of introducing water into the porous channels of the porous PFPE materials; this occurs despite the very hydrophobic nature of PFPE based materials.